

**EVALUATION OF REFORMULATED THERMAL CONTROL COATINGS
IN A SIMULATED SPACE ENVIRONMENT
PART I. - YB-71**

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ABSTRACT

The Air Force Space and Missile Systems Center and Wright Laboratory Materials Directorate (WL/ML) have sponsored an effort to reformulate and qualify Illinois Institute of Technology Research Institute (IITRI) spacecraft thermal control coatings.

S13G/LO-1, Z93, and YB-71 coatings were reformulated because the potassium silicate binder, Sylvania PS-7, used in the coatings is no longer manufactured. Coatings utilizing the binder's replacement candidate, Kasil 2130, manufactured by The Philadelphia Quartz (PQ) Corporation, Baltimore, Maryland, are undergoing testing at the Materials Directorate's Space Combined Effects Primary Test and Research Equipment (SCEPTRE) Facility operated by the University of Dayton Research Institute (UDRI). The simulated space environment consists of combined ultraviolet (UV) and electron exposure with *in situ* specimen reflectance measurements. A brief description of the effort at IITRI, results and discussion from testing the reformulated YB-71 coating in SCEPTRE, and plans for further testing of reformulated Z93 and S13G/LO-1 are presented.

INTRODUCTION AND BACKGROUND

The exterior surfaces of all spacecraft are covered with a combination of insulation and thermal control coatings to regulate the internal temperature of the spacecraft. This temperature depends on the internally generated heat of the spacecraft, the heat absorbed from the sun, and the heat radiated out to space. Insulation prevents heat from the sun from entering the spacecraft, but also prevents heat from escaping. Therefore, areas of the exterior surface must be exposed to space to allow radiation of excess heat. These areas, called radiators, are coated with thermal control coatings that allow radiation of thermal energy while reflecting most of the incident solar energy. The thermal balance of the spacecraft is determined by the solar absorptance (α_s) and the thermal emittance (ϵ) of this surface coating. Usually, the thermal control coatings are chosen to provide the lowest possible absorptance and the highest possible emittance, for a minimum α_s/ϵ ratio. In addition, the coatings must be easy to apply, durable, low outgassing and stable in the space environment.

Zinc orthotitanate (Zn_2TiO_4 , a.k.a., ZOT) and zinc oxide (ZnO) pigmented coatings are commonly utilized on spacecraft because of their optical properties and stability to the space environment. Three state of the art thermal control coatings, Z93, YB-71 and S13G/LO-1, supplied to the industry sole source by IITRI, incorporate potassium silicate as a major and vital component in their composition. Potassium silicate is manufactured using elevated temperatures and pressures to generate specific molar ratios of K_2O and SiO_2 (ref. 1) and is purchased in solution form. Upon drying, the potassium silicate becomes a mixture of primarily K_2O and SiO_2 , with other various silicates of potassium ($K_2Si_2O_5$, K_2SiO_3 , $K_2Si_4O_9 \cdot H_2O$, and $KHSi_2O_5$) theoretically present.

In the case of ZnO pigmented S13G/LO-1, which uses a silicone binder, the potassium silicate is used to reactively encapsulate the ZnO pigment particles. This allows for the formation of a barrier to photodeposition reactions on the surface of ZnO and enhances the space stability of the coating. In the case of the inorganic ZnO pigmented Z93 and ZOT pigmented YB-71, the potassium silicate constitutes the binder in the coatings. The potassium silicate used in the formulation of white spacecraft thermal control coatings, Z93, YB-71, and S13/GLO-1, was manufactured by Sylvania as PS-7 potassium silicate solution. Sylvania has discontinued the manufacture of PS-7, which has threatened the continued availability of these coatings to AF space systems. To ensure the continued supply of these thermal control coatings, WL/ML began a contracted effort with IITRI (points of contact: Yoshiro Harada and Mukund "Mike" Deshpande) in October of 1991 to reformulate and qualify new versions of Z93, YB-71, and S13G/LO-1 with an alternate and available binder system. The Air Force Space and Missiles Systems Center (SMC) sponsored this contractual effort and Jerry Bauer and Michael Meshishnek of The Aerospace Corporation provided technical guidance to the Materials Directorate.

IITRI identified The PQ Corporation's Kasil 2135 potassium silicate solution as an ideal replacement material for the Sylvania PS-7. The Kasil 2135 had better purity levels than the PS-7, but production of the Kasil 2135 is only performed on limited research levels, thus it was not considered a viable alternative. The PQ Corporation indicated that production of Kasil 2130 would be continued indefinitely. Since Kasil 2130 has identical SiO₂:K₂O mole ratio of 3.3:1 as PS-7 and similar purity levels, viscosity, and other properties, IITRI chose it as the candidate replacement binder early in their effort. IITRI used Kasil 2130 to reformulate the coatings, performed various processing and properties evaluation studies, and demonstrated that Kasil 2130 is a suitable candidate material to replace PS-7 in the three thermal control coatings. IITRI has designated the reformulated coating candidates as Z93P, YB-71P, and S13GP/LO-1. A Wright Laboratory technical report documenting the effort by IITRI will be available soon.

The final aspect of the program involves testing of the coatings in a simulated space environment and comparing the results to the original formulation coatings. Space simulation tests of these materials have been and are in the process of being conducted at various facilities. These include: UV & electron exposure testing at WL/ML's SCEPTRE facility and at The Aerospace Corporation, vacuum ultraviolet (VUV) & atomic oxygen exposure testing at both NASA Lewis and NASA Marshall, and planned UV & electron exposure testing at NASA Marshall. Additionally, the Canadian Space Agency flew samples of the candidate coatings in the "Materials Exposure in Low Earth Orbit (MELEO)" experiment on STS-52, where "all exposed samples showed no optical or physical degradation." IITRI has submitted coating samples to The Aerospace Corporation for flight testing on the upcoming Ballistic Missile Defense Organization (BMDO) sponsored Space Active Modular Materials Experiment (SAMMES).

The purpose of this study was to determine the performance of YB-71 and YB-71P in a multiple radiation environment consisting of high vacuum, low energy electron, and UV radiation. The results from the testing of Z93P and S13GP/LO-1 will be presented in the future.

SCEPTRE DESCRIPTION

WL/ML's Space Combined Effects Primary Test Research Equipment (SCEPTRE) Facility is the only Air Force owned facility designed specifically for testing and qualification of spacecraft thermal control coating materials. Testing at the facility is performed in accordance with the guidelines established by the American Society for Testing and Materials (ASTM) *E 512-94 Standard Practice for Combined, Simulated Space Environment Testing of Thermal Control Materials with Electromagnetic and Particulate Radiation* (ref. 2). The system has the capability of providing synergistic UV and electron radiation environments similar to those experienced by satellites orbiting in mid-to-high earth orbits and is shown in figure 1. In addition, the system has the ability to perform *in situ* measurements of sample temperature and reflectance as a function of wavelength. The vacuum level is maintainable from approximately 6.7×10^{-6} to 6.7×10^{-5} Pa (5×10^{-8} to 5×10^{-7} Torr), the

sample temperatures can range from room temperature to 100°C (212°F), the simultaneous multiple sun levels average between 1.0 to 3.0 equivalent ultraviolet suns (EUVS) (250-400 nm), and the electron flux is adjustable to a maximum of 10^{12} e⁻/cm²/sec at energies adjustable to a maximum of 20 KeVs, for each of the two electron guns. This combination of specifically tailored parameters provides an accelerated testing environment with synergistic effects of vacuum, accelerated UV and electron radiation, and limited thermal cycling.

Vacuum System

The vacuum system is composed of a 45.72 cm (18 in) diameter belljar pumped by a Welch Vacuum Technology model 3106S turbomolecular pump, backed by a Welch Vacuum Technology model 1397 rotary pump. The chamber is monitored via two different ion gages, a Granville-Phillips 271 Series and a Fredricks-Televac model 3C5-2A2; and a Uthe Technology Inc. (UTI) model 100C residual gas analyzer. The chamber has the capability of exposing a maximum of five, 2.38 cm (15/16 in) diameter, specimens to the synergistic UV and electron radiation. Figure 2. shows the facility's sample wheel, containing five white thermal control coating specimens, positioned just below the Faraday cup. The specimens are indirectly cooled via a chilled water line. The temperature control system reduces the exposed specimens' temperatures by approximately 5.56°C (10°F).

Solar Simulator

The solar simulator consists of a moderately filtered 2500 Watt xenon arc lamp mounted in a modified Spectrolab X-25 solar simulator. The entire optical system of the Spectrolab X-25 has been replaced and redesigned by the University of Dayton Research Institute. The xenon arc lamp is nominally filtered and is capable of generating 6 EUVS and has a non-uniform intensity distribution across the beam's profile, with the center of the beam being more intense than the edges. The output of the solar simulator is measured with an EG&G model 580 spectroradiometer that is calibrated using an EG&G 1000 Watt quartz-tungsten-halogen FEL style lamp (250-1100 nm) traceable to the National Institute of Standards and Technology (NIST) data.

Electron Guns

The SCEPTRE Facility utilizes two Kimball Physics Inc. EFG-11 electron guns. These guns are designed to provide a flood of electrons with energies ranging up to 20 KeV. Their output are monitored by a Faraday cup situated 6.98 cm (2.75 in) above the center most sample position (see figure 2). A Keithley 617 programmable electrometer is used to measure the electron current generated by the Faraday cup. The electron beam is adjusted using a phosphor screen and assumed to be relatively uniform across the specimens.

Spectrophotometer

The spectrophotometer used to monitor the specimens' solar absorptance is a Perkin-Elmer Lambda 9 UV-Vis-NIR double beam spectrophotometer and is fiber-optically coupled to a Labsphere Inc. integrating sphere located inside the vacuum chamber. The spectrophotometer is designed to provide *in situ* NIST traceable reflectance measurements and is utilized periodically throughout the duration of a test.

Data Acquisition

The data acquisition is performed by a Digital Equipment Corp. (DEC) VAXstation III/GPX, in conjunction with hardware from a variety of other vendors, and utilizes IEEE-488, RS-232, analog-to-digital, and digital-to-analog interfaces. The system monitors the electron flux, vacuum level, specimen backside temperatures, and residual gases present in the vacuum chamber every 15 seconds and periodically records the data throughout the duration of a test. It also acquires data from the spectrophotometer and spectroradiometer and is used to analyze and graphically display the data.

TEST DESCRIPTION

Testing of the YB-71 and YB-71P materials was intended to compare their relative performance in the SCEPTRE simulated space environment. Additionally, extra Z93 specimens were used as controls for direct comparisons between the three tests necessary for the reformulated IITRI materials. Each of the three tests was designed to have 1000 hours of exposure with accelerated UV and electron radiation. Due to the non-uniform distribution of the beam intensity described above, the level of UV acceleration varies with sample position in the SCEPTRE facility. The targeted UV levels for the five samples were: 3.0 EUVS for the Z93 control specimen and two specimens of each of the YB-71 and YB-71P materials each receiving 2.5 and 1.0 EUVS. The electron exposure is designed to provide accelerated low energy electron radiation compared to that experienced in geosynchronous orbits.

For the two electron guns that the facility has, energy levels of 1 KeV and 10 KeV were chosen. At the geosynchronous orbits there are both trapped and solar wind electrons. The AE8-MAX trapped electron model (ref. 3) and data originating from the SCATHA space flight experiment (ref. 4) were used in establishing the values of $3 \times 10^9 \text{ e}^-/\text{cm}^2/\text{sec}$ for the 10 KeV electrons and $6 \times 10^9 \text{ e}^-/\text{cm}^2/\text{sec}$ for the 1 KeV electrons. At 1000 hours of exposure this would generate a total fluence of $3.24 \times 10^{16} \text{ e}^-/\text{cm}^2$. Reference 4 indicates that the fluence of solar plasma electrons within the energy range of 0-10 KeV would be $5.8 \times 10^{15} \text{ e}^-/\text{cm}^2$ in a 1000 hour period, therefore the acceleration factor for the electron exposure is 5.5.

SCEPTRE RESULTS

The first 600 hours of the YB-71 test, number 93QV01, proceeded very well. However, at 602 hours into the test, a power outage occurred that lasted approximately 40 minutes. This power outage caused a temporary loss in vacuum (estimated to be $1.0 \times 10^{-3} \text{ Pa}$ ($7.5 \times 10^{-3} \text{ Torr}$)) and ultimately caused the electron gun filaments to burn out. The test was continued to 1000 hours of exposure with UV radiation only. The performance of the YB-71P material was very disappointing. Very early in the test it was obvious that the YB-71P material was not performing nearly as well as the YB-71. Figure 3. shows the results of the simulated space environment on all the Z93, YB-71, and YB-71P specimens' solar absorptance. All data in this graph were obtained by *in situ* reflectance measurements, except the last data points, which were measured within a few hours after exposure to air. These results were not satisfactory and created a lot of concern.

Synergistic atomic oxygen and VUV exposure testing performed at NASA Lewis (ref. 5), had showed Z93P to be a poor performer on one occasion. These results also generated a lot of concern, so the Z93P was re-tested with a specimen from a different batch of material. The re-test found the problem to be isolated to the batch of coating tested. For this reason it was thought that there might be a similar problem with a batch of the YB-71P. Since both the poor performing Z93P (tested at NASA Lewis) and the poor performing YB-71P (tested with SCEPTRE) were made with the same lot of Kasil 2130, it was decided to re-test the YB-71 and YB-71P in SCEPTRE. The second test, number 93QV02 exposed a YB-71 specimen, a YB-71P "bad batch" specimen (which was used as a reference in the 93QV01 test), two YB-71P specimens from a different batch, and a Z93 cross-reference specimen.

Performance of the second test was affected due to optical alignment problems within the solar simulator. The situation limited the unobscured UV exposure to three sample positions while the two outer-most positions were exposed to an unknown amount of gradient shading. Thus, the cross-reference Z93 specimen was not used in the same position as the 93QV01 test and only three specimens: the YB-71, YB-71P (suspected bad batch), and YB-71P (new batch), were exposed to known UV levels. The UV exposure of the second YB-71P (new batch) and the Z93 cross-reference specimens were not discernible, but were estimated to be about 1.0 EUVS.

The re-test of YB-71P showed that there was a difference in performance of the two different batches of YB-71P. Figure 4, shows the comparison of the YB-71 and YB-71P performance during the 93QV02 and 93QV01 tests. As previously mentioned for the 93QV01 data, all the 93QV02 data points were measured in vacuum, except the last 3 points which were measured in air over a period of 12 days. This figure shows that the new YB-71P can perform identically to the old YB-71 material and verifies the repeatability of results for like materials from different SCEPTRE tests. Histories for the 93QV01 and 93QV02 tests, including specimen backside temperature (figures 5-6), electron flux (figures 7-8), and specimen spectral reflectance data (figures 9-16) are included for reference. Note that in these figures, the suspected bad batch of YB-71P is labeled with an asterisk (YB-71P*). A summary of the test environments and solar absorptance data for each specimen, from both tests, is provided in table 1.

As mentioned above, there was a difference, between the tests, in the specimens' rates of solar absorptance "recovery" after exposure to air. The difference can only be attributed to the fact that after the second test, 93QV02, the specimens were not directly brought back to ambient conditions but the vacuum chamber was first purged with gaseous nitrogen prior to atmospheric exposure. This resulted in the specimens taking more than 12 days to recover only a fraction of their reflectance instead of essentially recovering instantaneously as they did in the 93QV01 test, without the nitrogen purge. It can be assumed that the gaseous nitrogen molecules, while not chemically reacting with the materials, did physically block access of the oxygen atoms to the material and restricted the rate and even extent of the oxygen recombination process that restores some of the material's reflectance properties.

Visual inspection of the specimens revealed that the old formulation of YB-71 tested in the 93QV02 test had a non-uniform, "blotchy" pattern of light and dark areas within the coupon. The poorer performing specimens from both tests exhibited more crazing (or micro-cracking) due to the shrinking associated with the loss of moisture from the materials than did the other specimens. The poorer performing specimens also exhibited a small amount of adhesion loss near the edges of the coupon.

Material	EUVS (250-400 nm)	1 KeV fluence (e-/cm2)	10 KeV fluence (e-/cm2)	Pretest solar absorptance (in vacuum)	Posttest solar absorptance (in vacuum)	Change in solar absorptance (in vacuum)
93QV01 (1000 hrs.)						
Z93	3.2	1.66E+16	8.32E+15	0.135	0.185	0.050
YB-71	2.6	1.66E+16	8.32E+15	0.087	0.232	0.145
YB-71	1.7	1.66E+16	8.32E+15	0.090	0.220	0.130
YB-71P*	2.1	1.66E+16	8.32E+15	0.089	0.282	0.193
YB-71P*	2.9	1.66E+16	8.32E+15	0.092	0.363	0.271
93QV02 (1265 hrs.)						
Z93	1.2 (est.)	2.03E+16	1.01E+16	0.134	0.178	0.044
YB-71	2.8	2.03E+16	1.01E+16	0.097	0.297	0.200
YB-71P*	2.7	2.03E+16	1.01E+16	0.106	0.419	0.313
YB-71P	2.5	2.03E+16	1.01E+16	0.093	0.266	0.173
YB-71P	1.1 (est.)	2.03E+16	1.01E+16	0.097	0.233	0.136

*suspected bad batch

Table 1. Summary of SCEPTRE Tests 93QV01 & 93QV02

XPS ANALYSIS

Several weeks after the completion of the 93QV01 test, surface analysis (X-ray Photoelectron Spectroscopy, XPS) was performed on the exposed YB-71P specimens, using a Surface Science Instruments apparatus which

utilizes a monochromatic X-ray source and charge neutralization, to determine if their poor performance was due to contamination. The XPS did reveal a slight amount of carbon but not enough to attribute to significant amounts of contamination.

Prior to the 93QV02 test, XPS was performed on a YB-71 specimen (which ended up being used as a reference in the 93QV02 test) and a YB-71P specimen from the suspected bad batch (which was exposed in the 93QV02 test) that showed a difference between the composition of the YB-71 and the YB-71P materials. The suspected bad batch YB-71P specimen contained more potassium than did the YB-71 specimen. The difference was measurable but initially not thought significant.

After the 93QV02 test, all three exposed YB-71 and YB-71P specimens and the exposed Z93 specimen were examined with the XPS analysis. This analysis showed that both the YB-71 and new batch of YB-71P contained about 12.5% potassium while the “bad batch” of YB-71P contained about 20% potassium. The Z93 specimen showed the smallest amount of potassium of any of the IITRI materials analyzed so far. This is interesting because, this material was shown to be the best performer in the 93QV01 test. Additionally, the light and dark areas of the “blotchy” areas previously discussed on the YB-71 used in the second test were separately examined with XPS. The results of this analysis are extremely interesting because it showed that in the old formulation of YB-71, with the Sylvania PS-7 binder, that the light areas contained 13.6% potassium and the darker areas contained 17.9% potassium. Thus, the XPS analysis suggests that there is direct link to the coating’s performance and its potassium concentration at the surface. Table 2. summarizes the XPS results.

Material	K	Si	K:Si Ratio	Change in solar absorbance (in vacuum)
93QV01 (1000 hrs.)				
YB-71P* (2.85 EUVS)	22.2%	9.2%	2.41	0.271
YB-71P* (2.07 EUVS)	21.0%	9.8%	2.14	0.193
93QV02 (1265 hrs.)				
YB-71P*				
pretest	20.3%	9.9%	2.05	*****
posttest (2.70 EUVS)	20.4%	9.9%	2.06	0.313
Z93 (1.2 EUVS, est.)	12.3%	22.1%	0.56	0.044
YB-71P (2.50 EUVS)	12.6%	16.0%	0.79	0.173
YB-71 (2.80 EUVS) (darker area)	17.9%	12.0%	1.49	*****
YB-71 (2.80 EUVS) (lighter area)	13.6%	15.8%	0.86	*****

* suspected bad batch

Table 2. Summary of XPS Results

RESULTS & DISCUSSION

As mentioned in the Introductory section, the SiO₂:K₂O mole ratio of both PS-7 and Kasil 2130 is 3.3:1. This yields a theoretical K:Si ratio for the coatings of 0.6061. XPS analysis reveals that the YB-71P coating that performed well had a K:Si ratio close to the theoretical value. However, the poor performing coatings possessed significantly higher K:Si ratios. It is hypothesized that the excess potassium is the cause in the increased degradation of the coatings. Additional analysis needs to be performed to determine if the cause of the excess potassium is from the raw material itself (i.e., poor quality control of K/Si ratio) or from processing/ application parameters (humidity, moisture content, temperature, etc.) at IITRI. In order to help answer this, additional XPS

analysis is planned to determine if the excess potassium is found uniformly through the depth of the coating or only at the surface.

The reflectance spectra for all the poor performing YB-71P specimens (figures 9, 10, and 14), while showing total overall reduction in reflectance, also show a depression in the reflectance curve around 950 nm that is more pronounced than in the YB-71 specimens (figures 12, 13, and 15). This absorption band, which is characteristic of Zn_2TiO_4 , is discussed in detail in a 1971 American Institute of Aeronautics and Astronautics (AIAA) paper authored by Zerlaut, *et. al.* (ref. 6). This paper describes the Ti^{+3} color center formation damage mechanism for Zn_2TiO_4 and presented findings which showed that reactively encapsulating the Zn_2TiO_4 with potassium silicate greatly reduces the extent of damage around 950 nm. The fact that the YB-71P coating is demonstrating a depression in the reflectance curve similar to that described in the paper is very interesting and presently unexplainable.

The inconsistency of the potassium silicate binder is a cause for concern. Similar testing of YB-71 and YB-71P materials from the same batches tested in SCEPTRE have been performed at The Aerospace Corporation, El Segundo, California. This testing has revealed similar results for the materials' performances. Currently, at the time of writing, testing is being performed at The Aerospace Corporation with 18 different samples of YB-71 and YB-71P materials to try to determine if the inconsistency is wide-spread or if this was just an isolated case.

Additional attempts at finding a replacement for the Sylvania PS-7 potassium silicate for use in other coatings, have been made by researchers at the Jet Propulsion Lab (JPL). They tried to identify a suitable conductive coating for use on the Cassini satellite's high gain antenna system and considered the NS43G coating (obtained from Space Control Coatings, Inc.), using both the PS-7 binder and the Kasil 2135 binder from The PQ Corporation as a possible candidate (ref. 7).

FUTURE PLANS

Further investigation is currently on going, in cooperation with IITRI and The Aerospace Corporation, to determine the extent of the variation of YB-71P material. The results from SCEPTRE testing of Z93P and S13GP/LO-1 will be presented in the future.

CONCLUSIONS & RECOMMENDATIONS

SCEPTRE testing of reformulated YB-71P was performed. Issues have risen concerning the reproducibility of the coating. Batch to batch variations in the stability of the coatings to the combined space environment were demonstrated. Some YB-71P specimens performed identically like the original YB-71 formulation. A different lot with a higher K:Si ratio performed very poorly. Additional testing of YB-71P is recommended to determine the cause of the batch to batch variations. It is suggested that lot testing of YB-71P coatings by the users be performed until the cause of the coating non uniformity can be determined.

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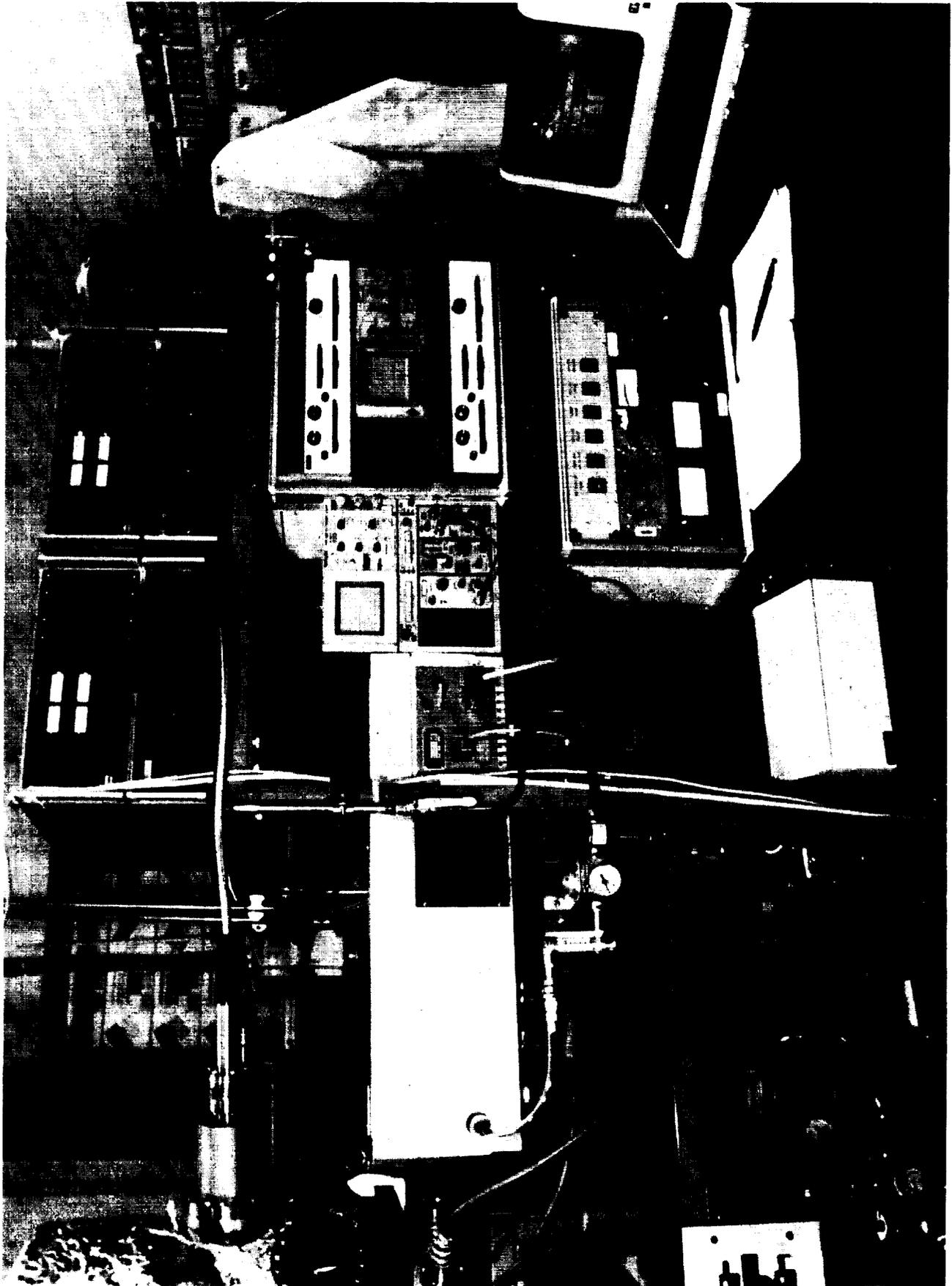


Figure 1. USAF WL/ML SCEPTRE Facility



Figure 2. SCEPTRE Facility's Sample Wheel and Faraday Cup

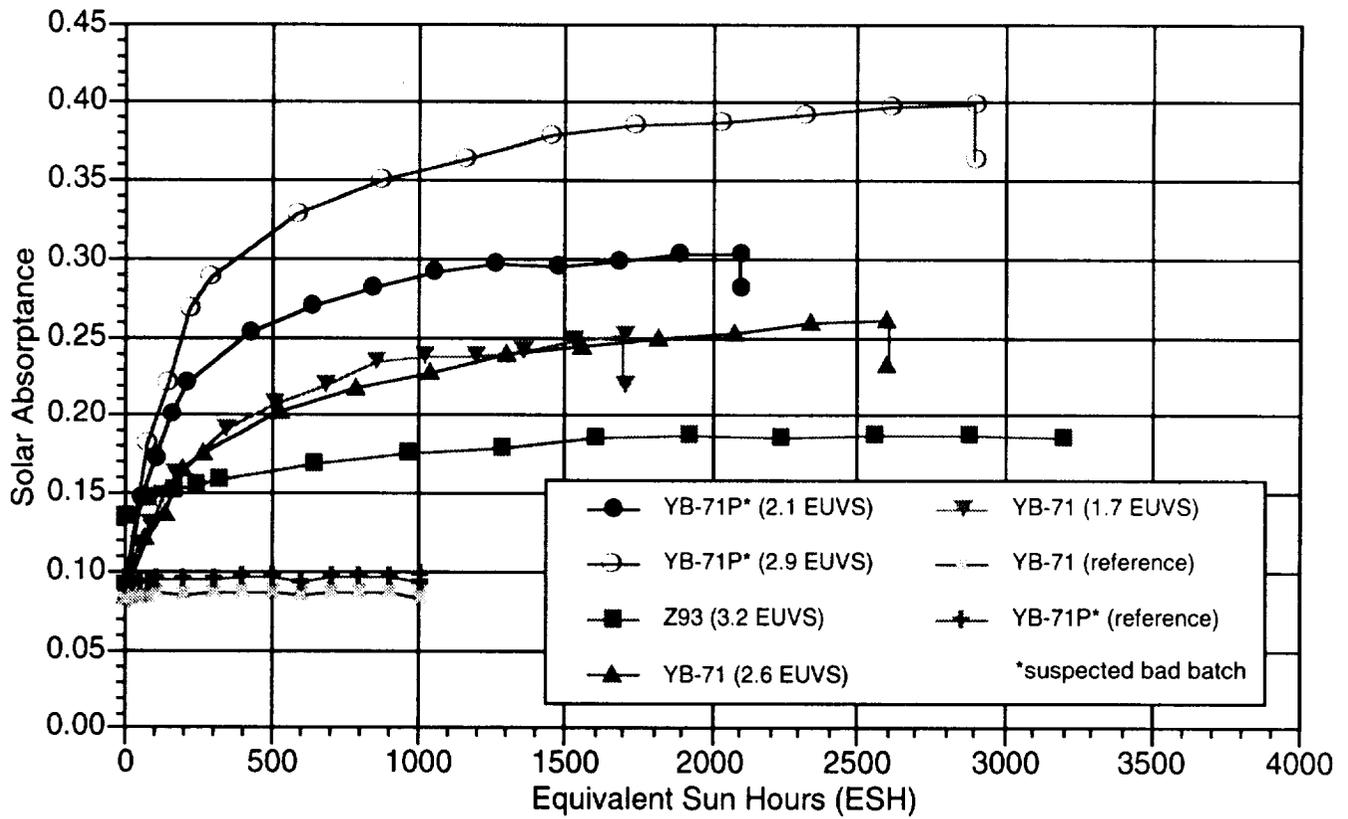


Figure 3. USAF WL/ML SCEPTRE Test 93QV01 Solar Absorbance History

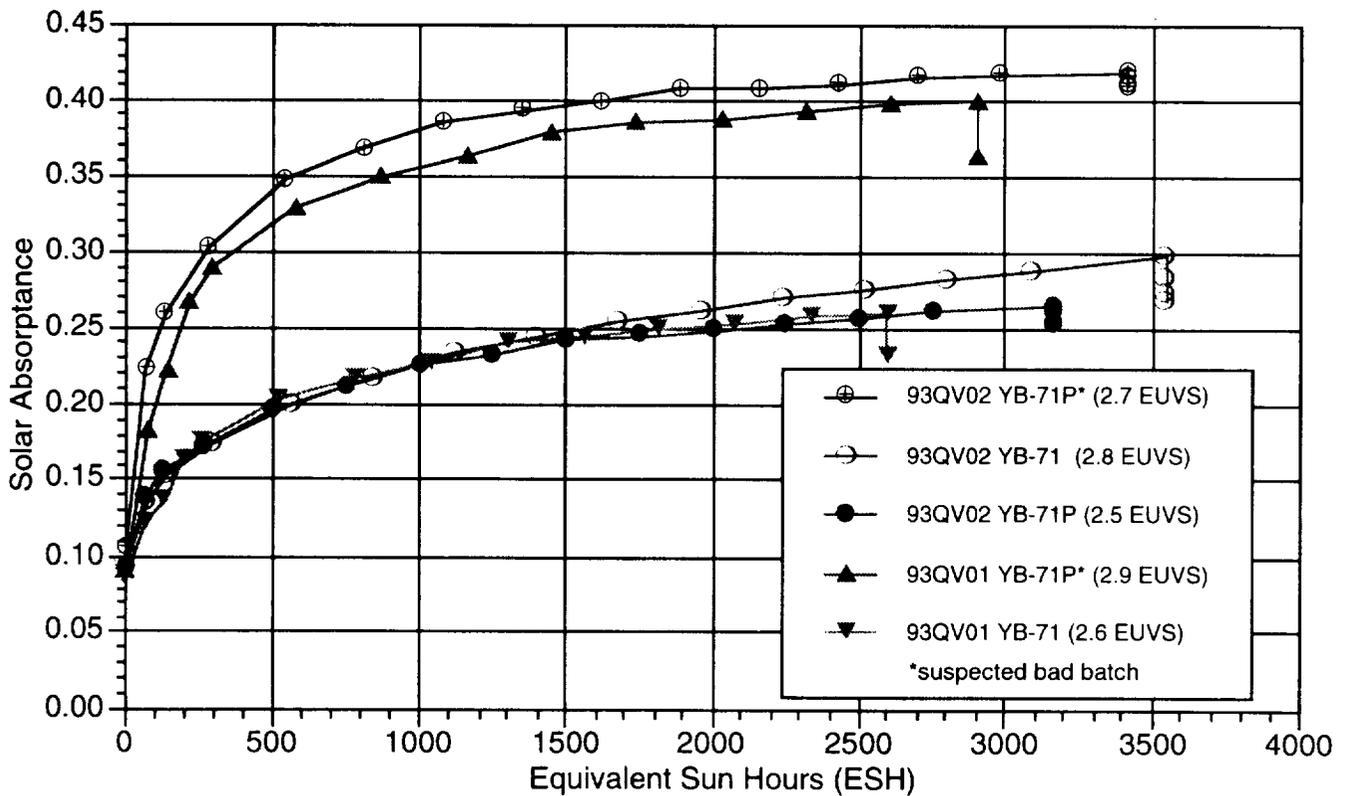


Figure 4. USAF WL/ML SCEPTRE YB-71 Requalification Results

Figure 5. SCEPTRE Test 93QV01 Specimen Temperature History

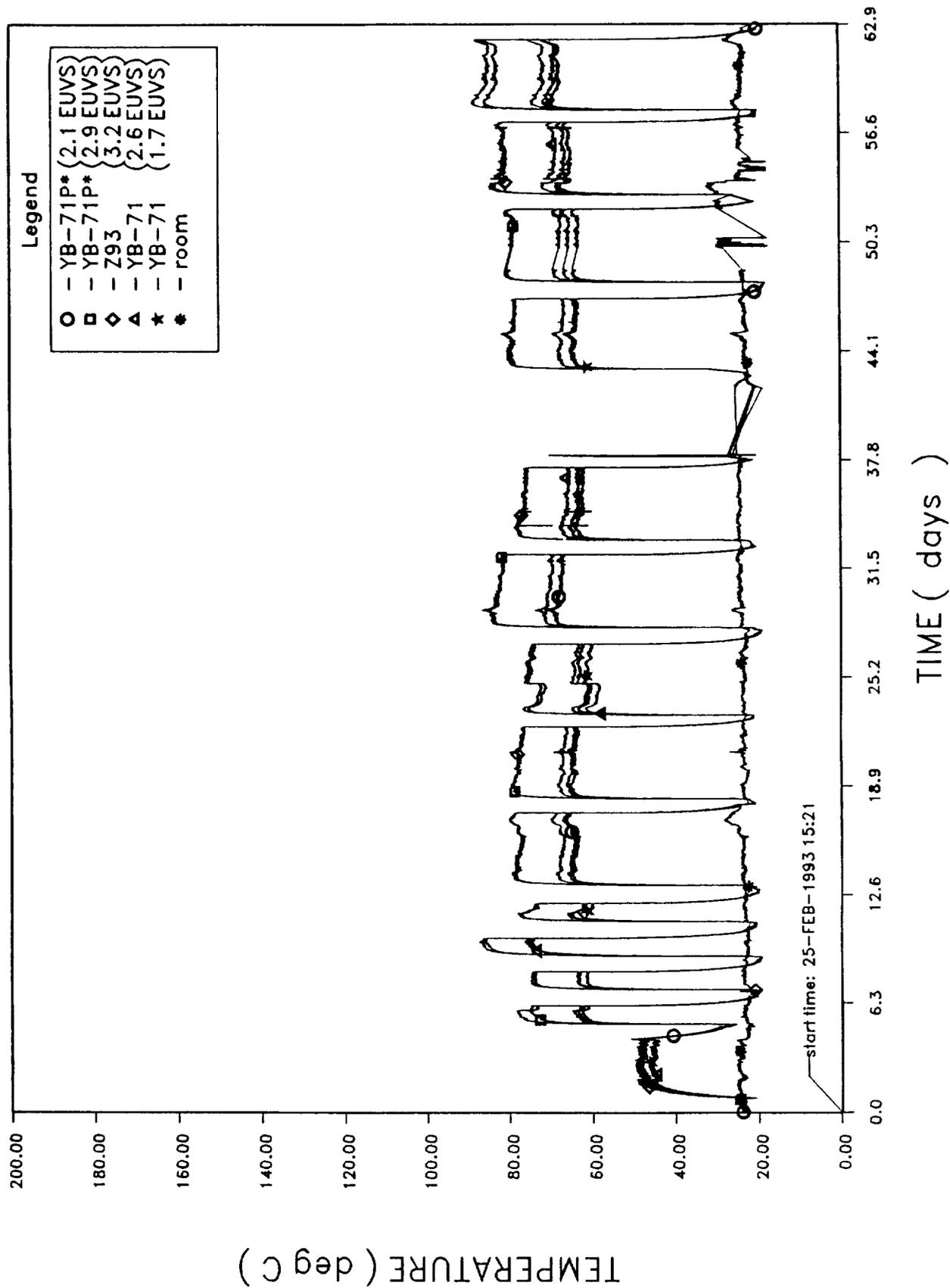


Figure 6. SCEPTRE Test 93QV02 Specimen Temperature History

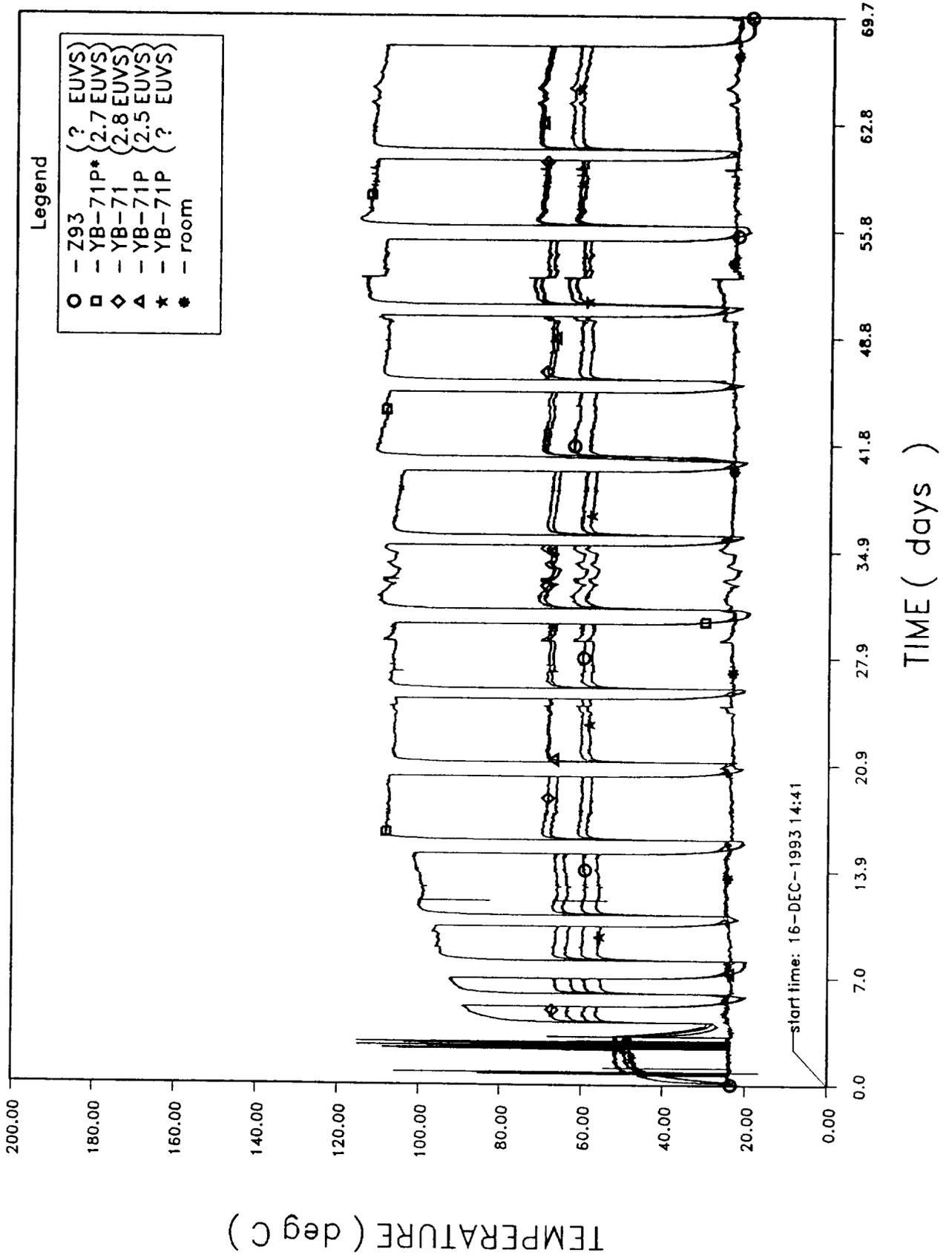


Figure 7. SCEPTRE Test 93QV01 Electron Flux History

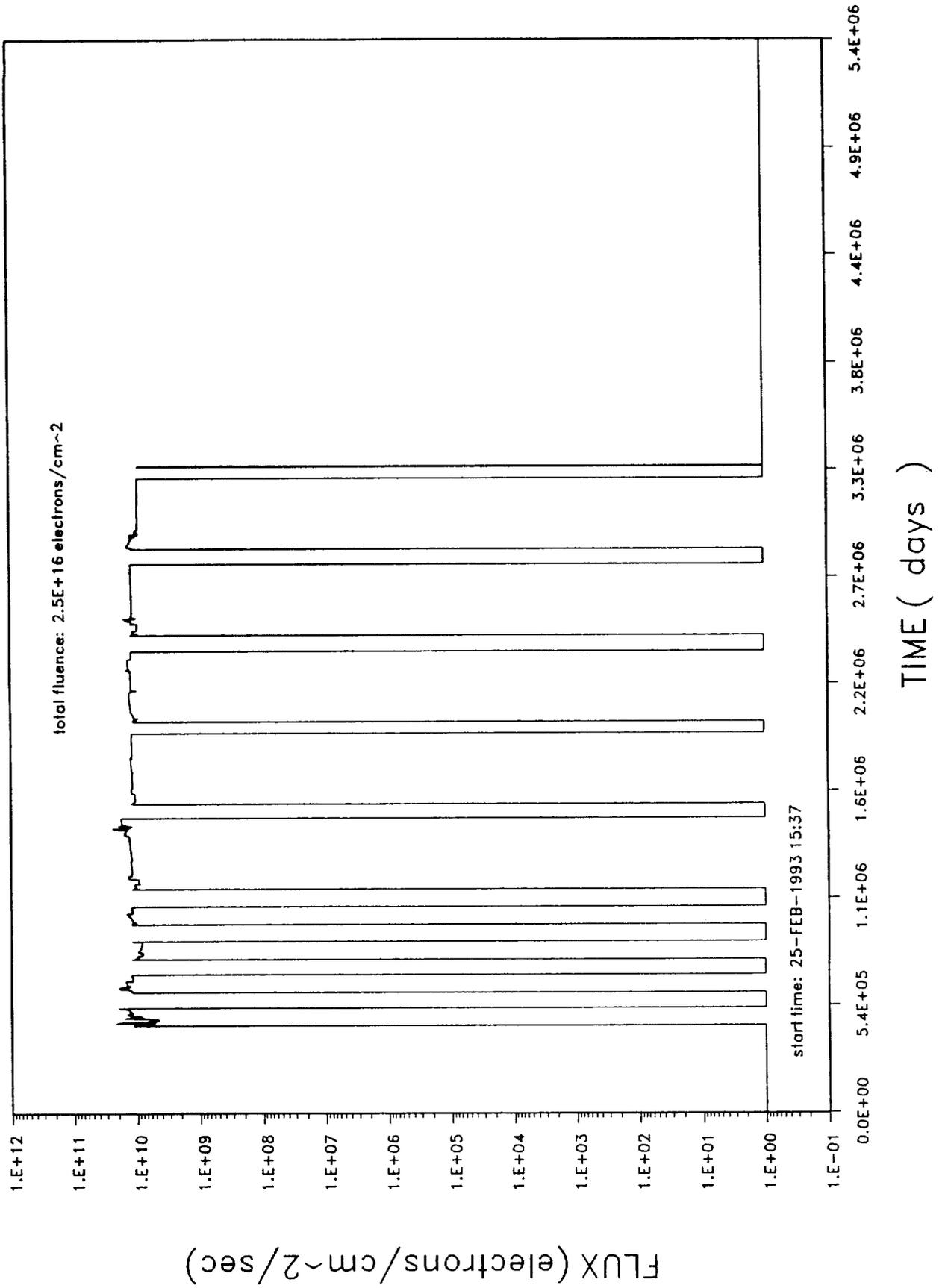


Figure 8. SCEPTRE Test 93QV02 Electron Flux History

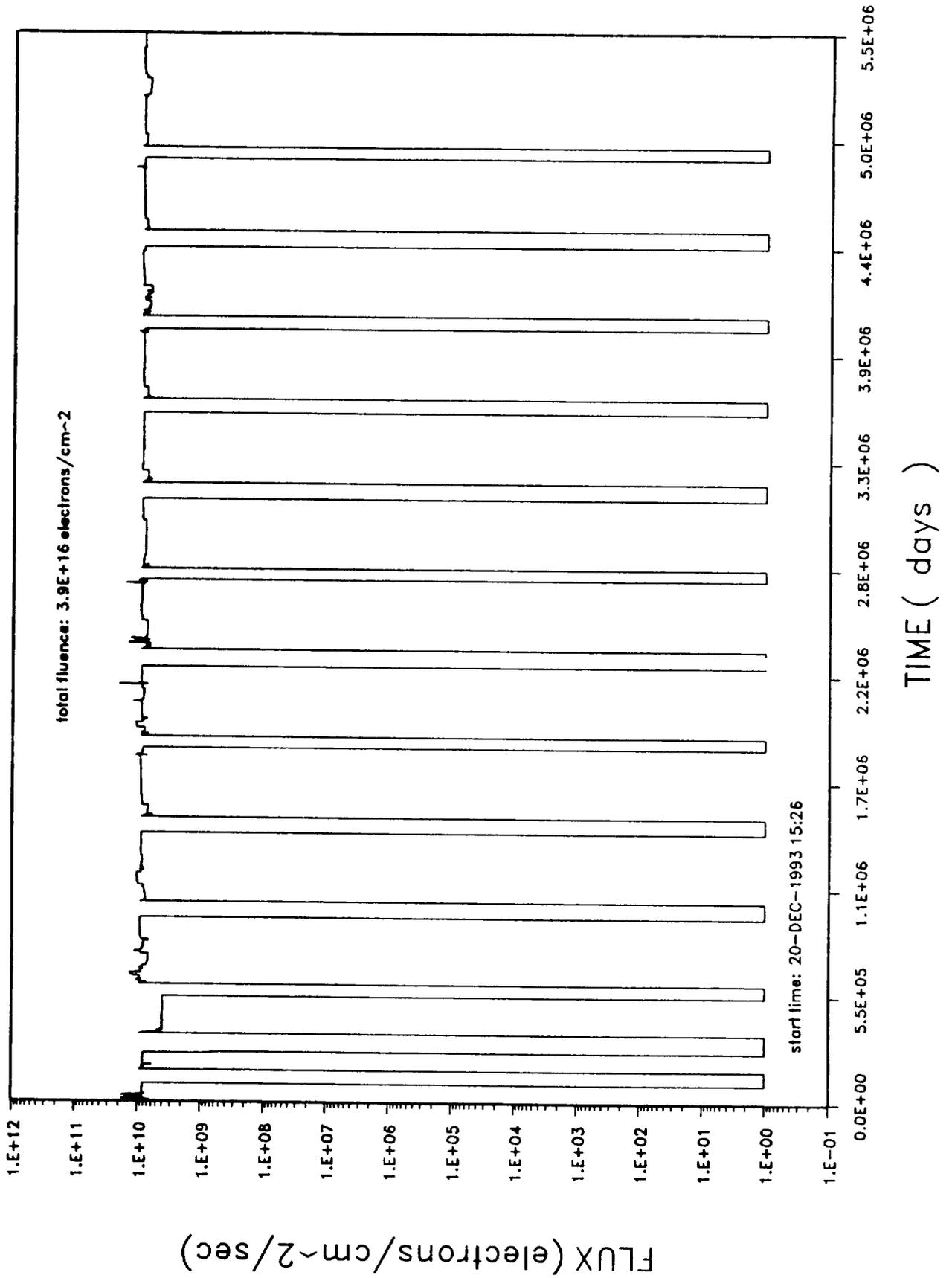


Figure 9. 93QV01 YB-71P* (2.1 EUVS) Reflectance History

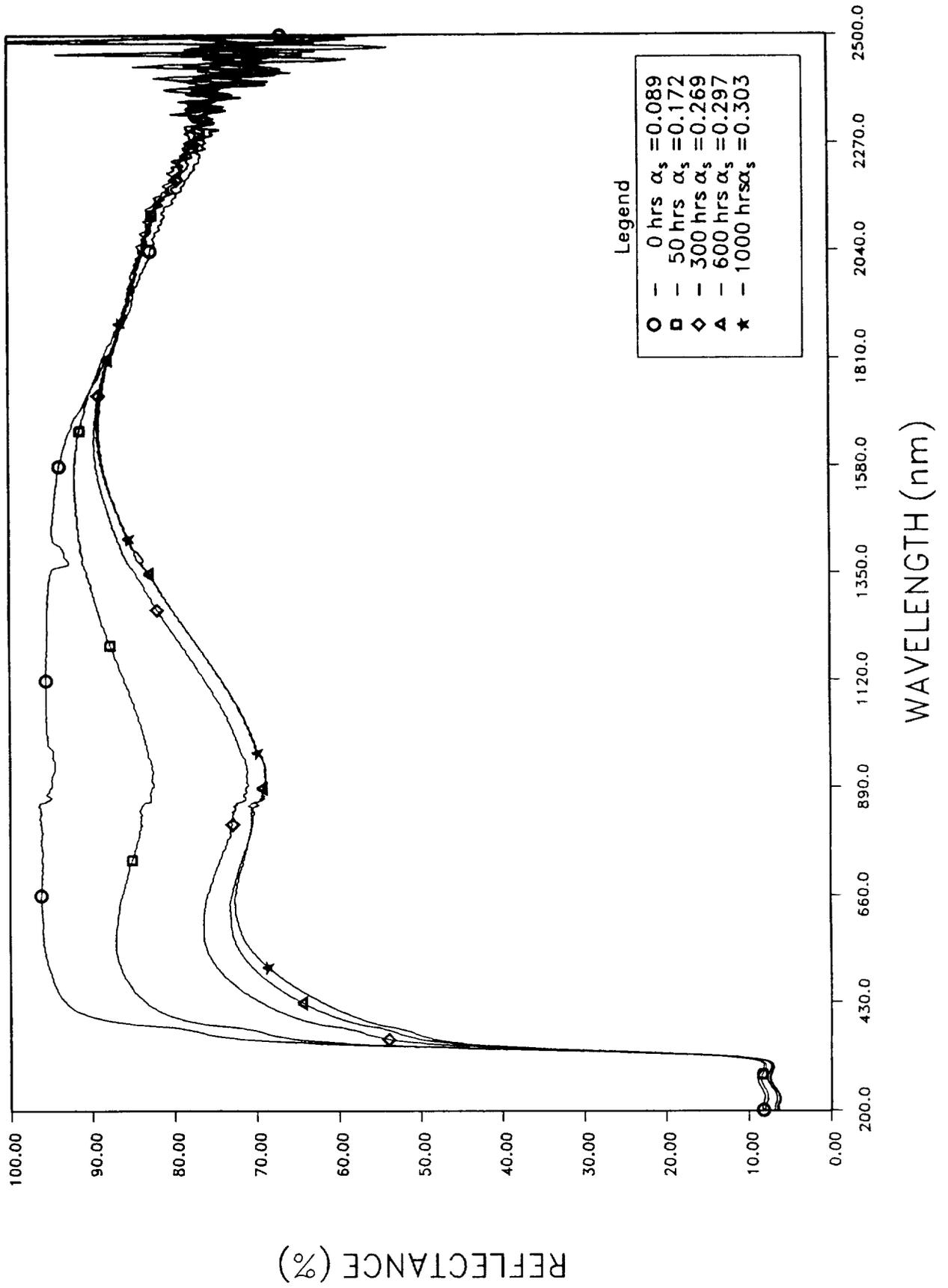


Figure 10. 93QV01 YB-71P* (2.9 EUVS) Reflectance History

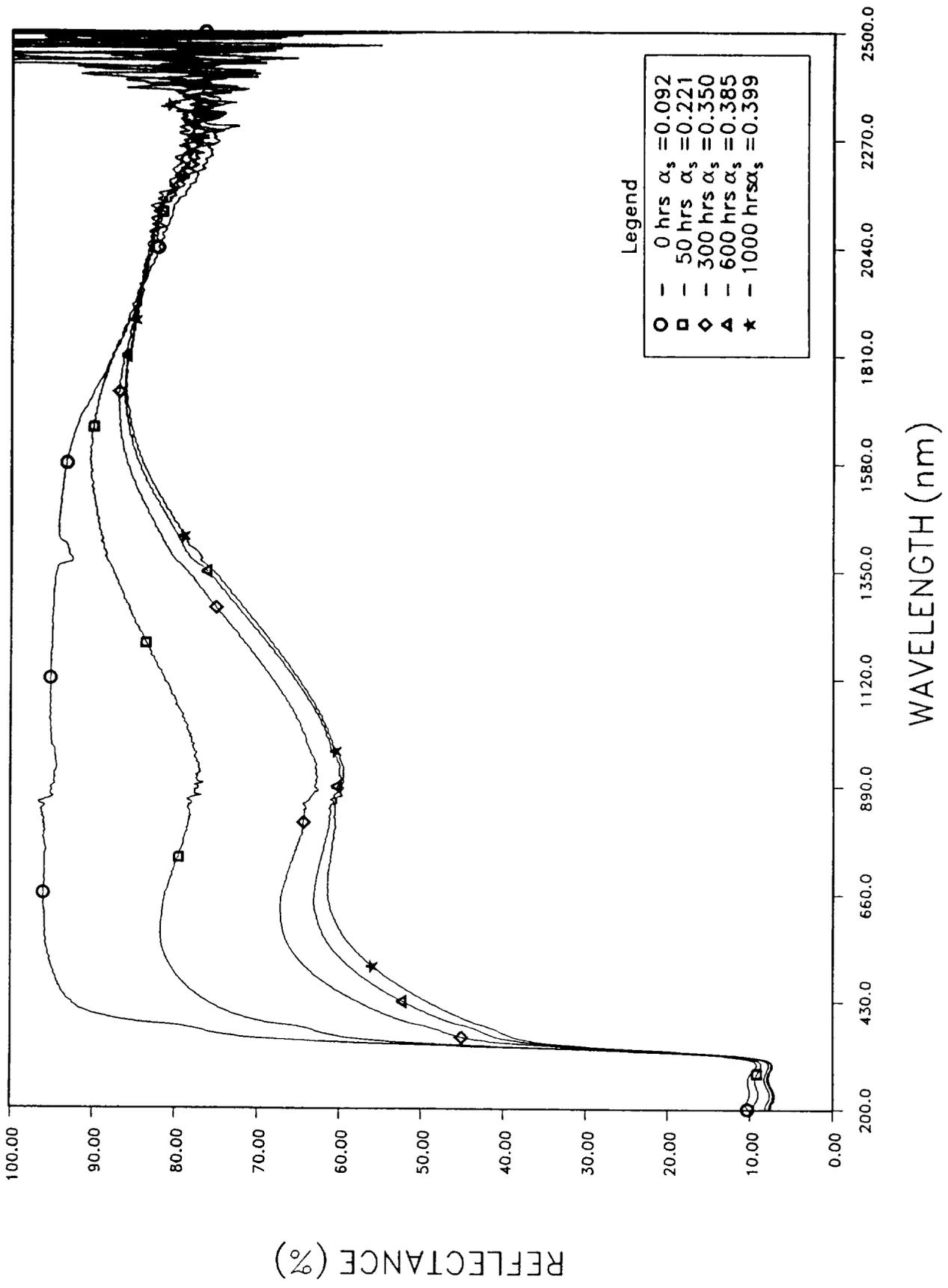


Figure 11. 93QV01 Z93 (3.2 EUVS) Reflectance History

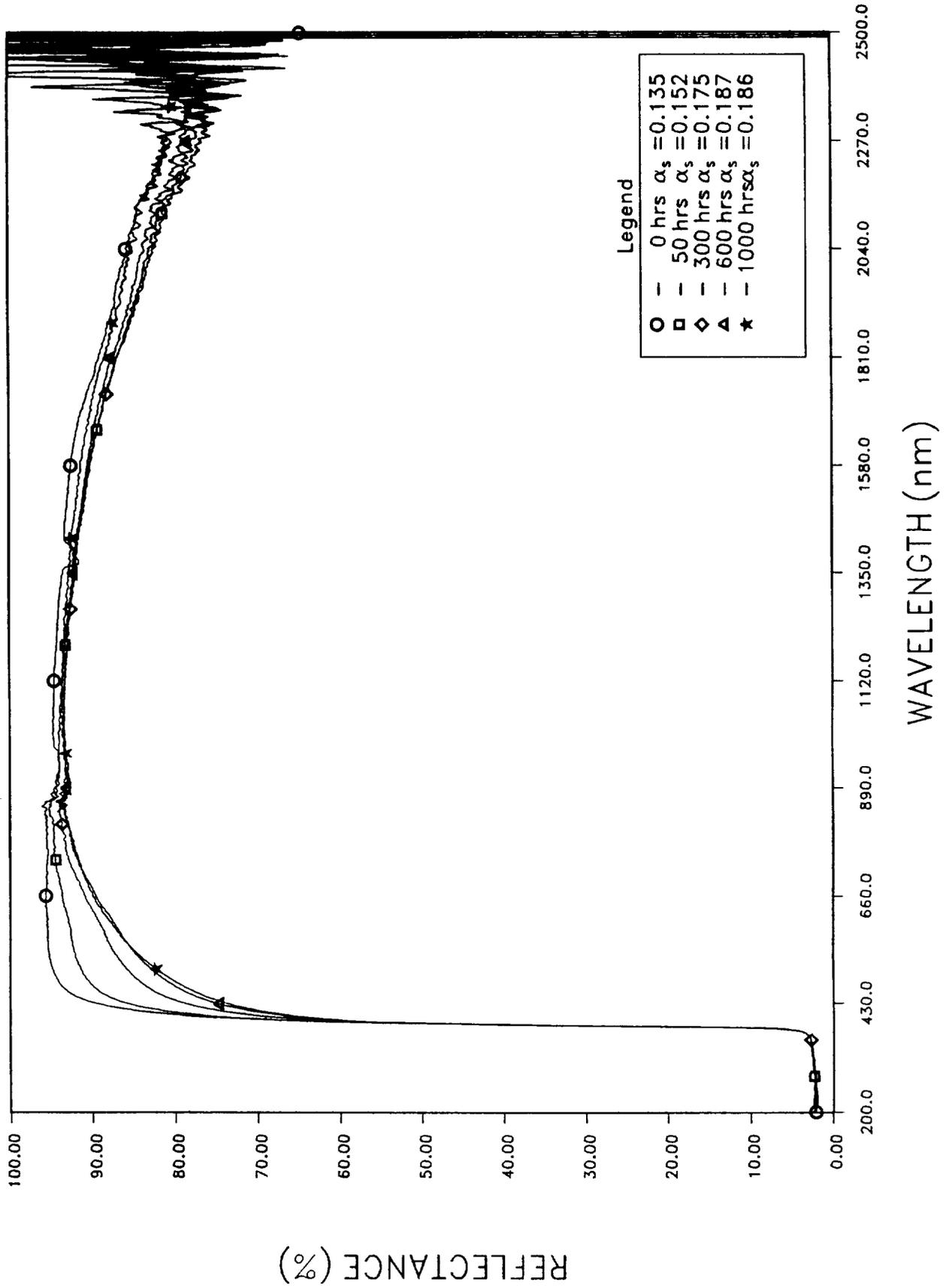


Figure 12. 93QV01 YB-71 (2.6 EUVS) Reflectance History

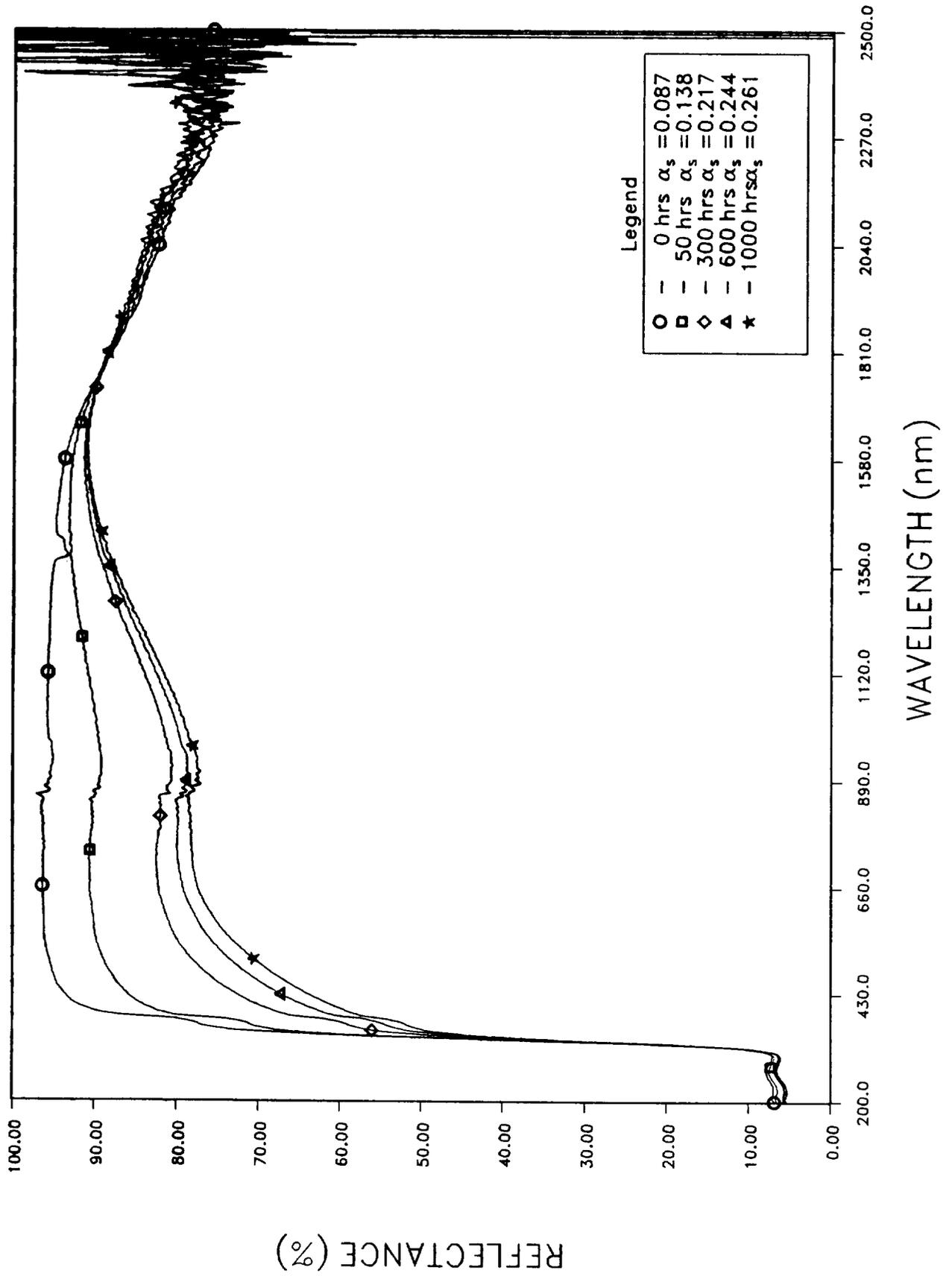


Figure 13. 93QV01 YB-71 (1.7 EUVS) Reflectance History

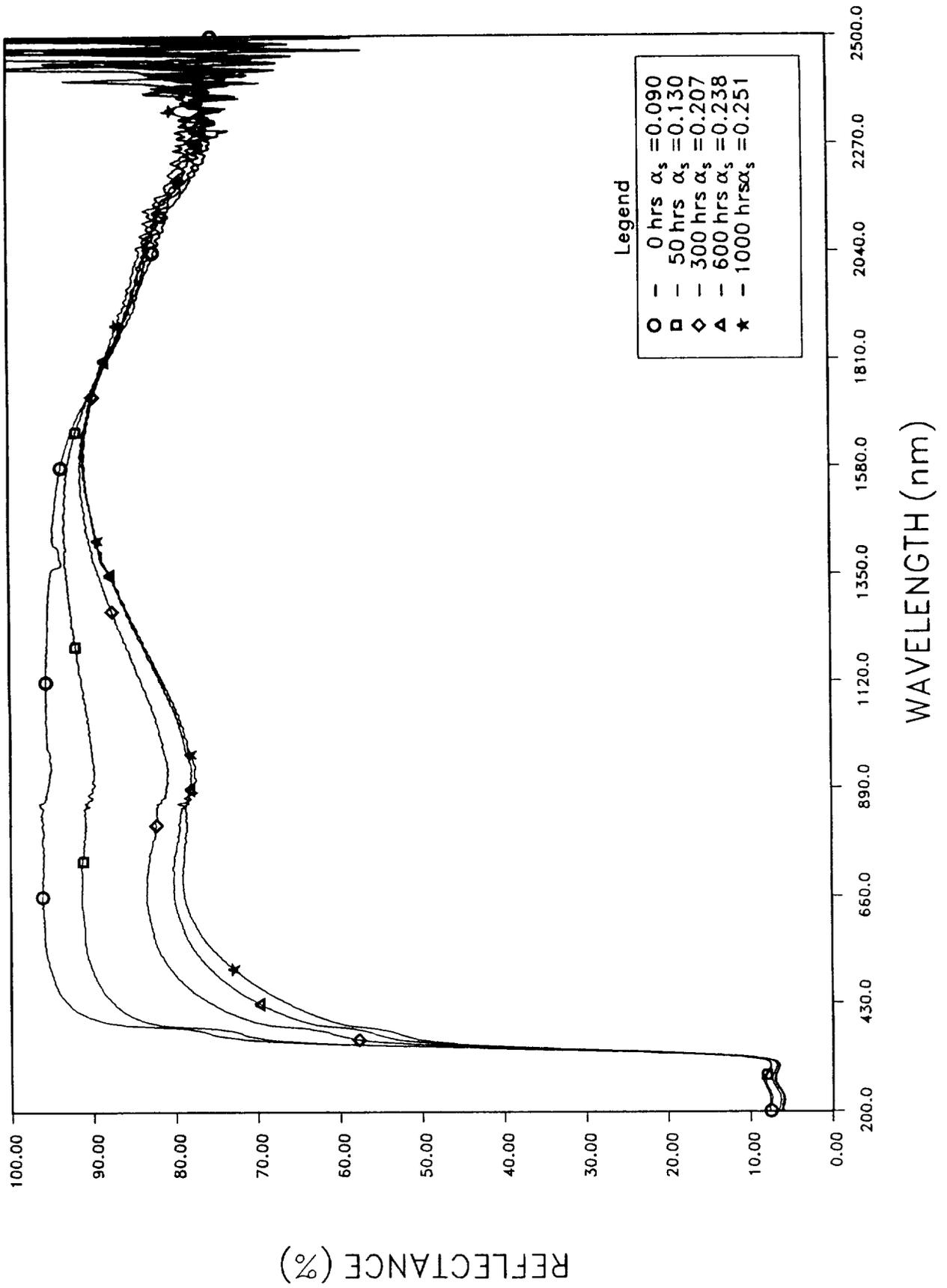


Figure 14. 93QV02 YB-71P* (2.7 EUVS) Reflectance History

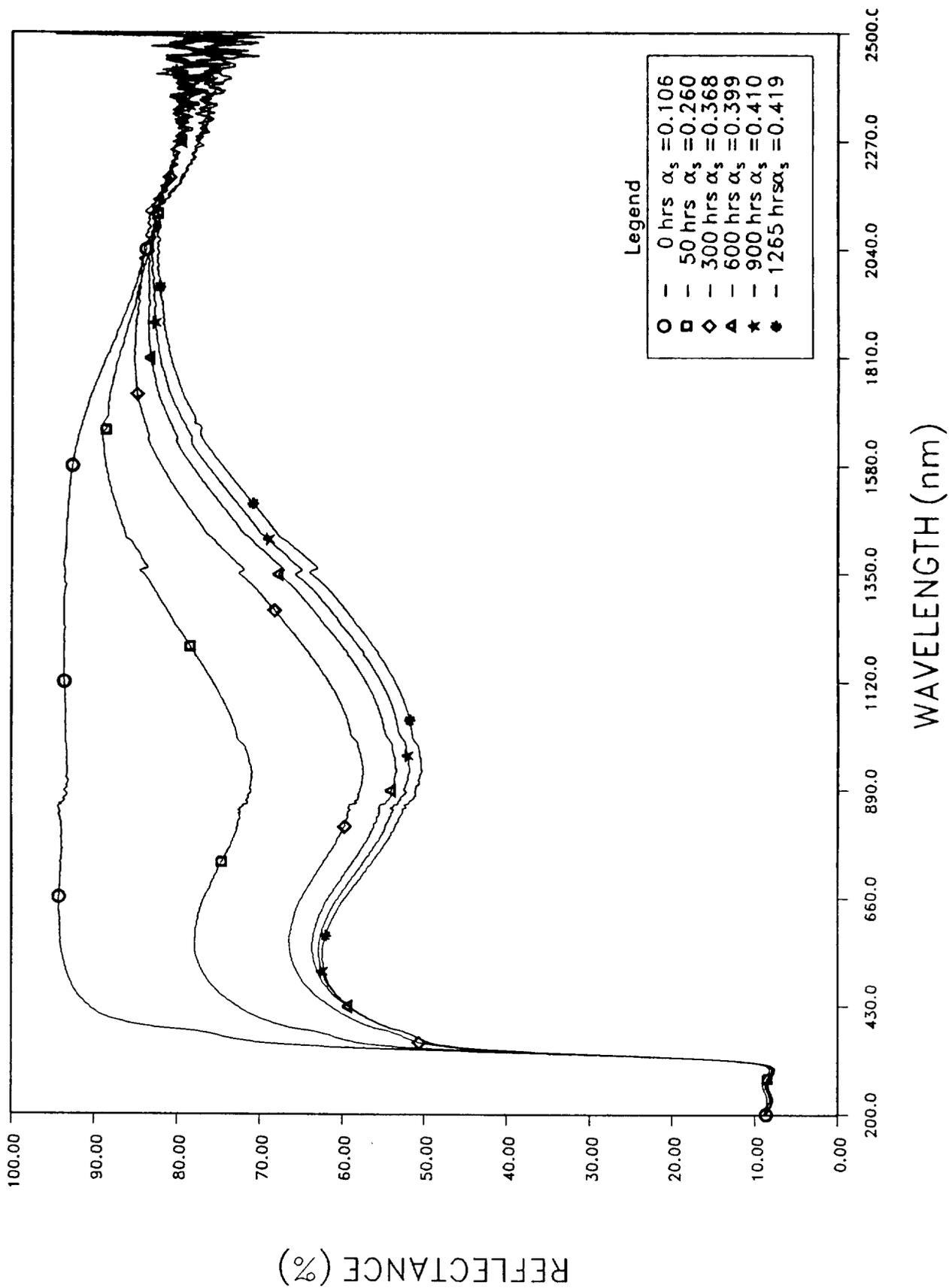


Figure 15. 93QV02 YB-71 (2.8 EUVS) Reflectance History

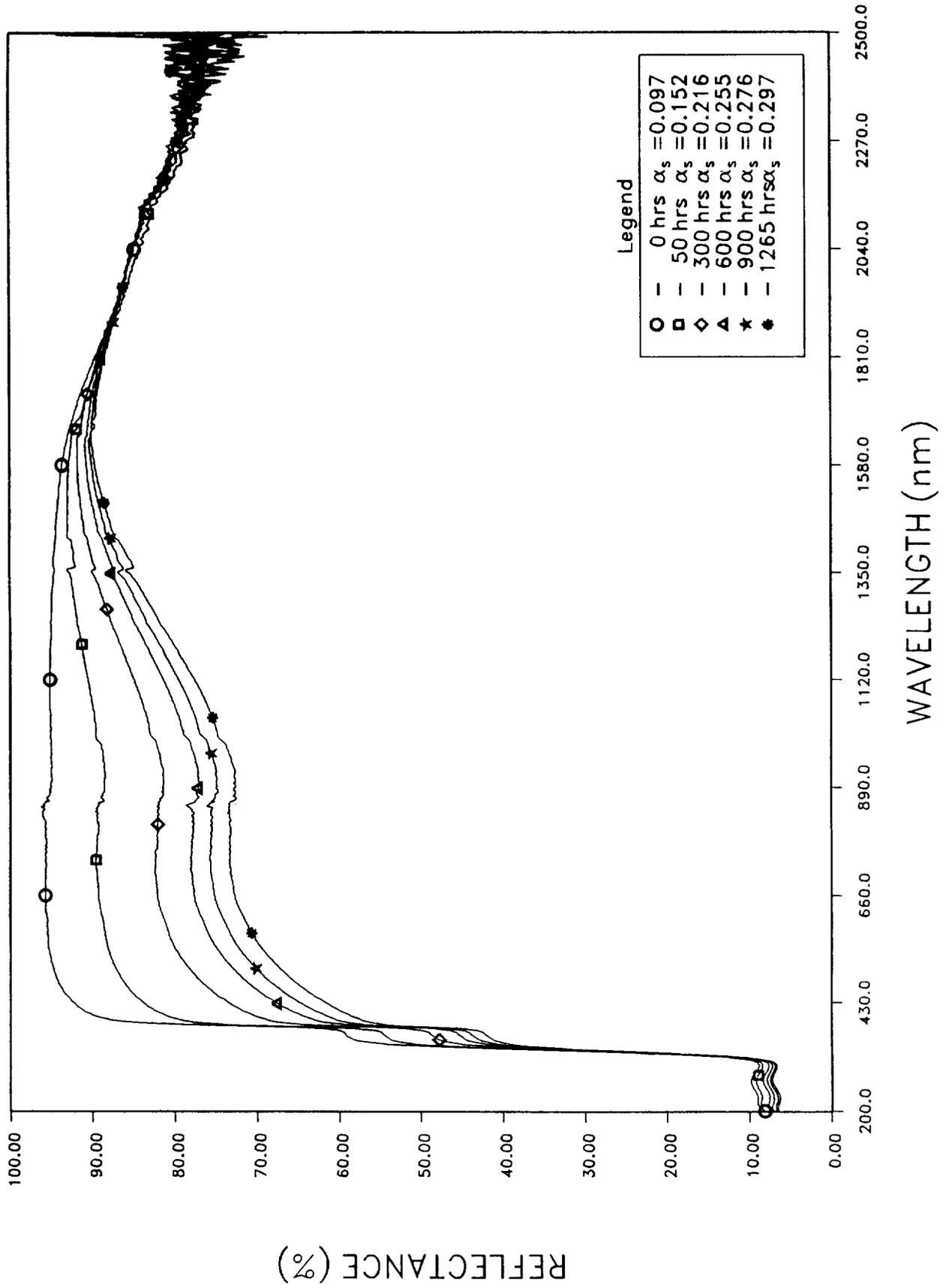


Figure 16. 93QV02 YB-71P (2.6 EUVS) Reflectance History

